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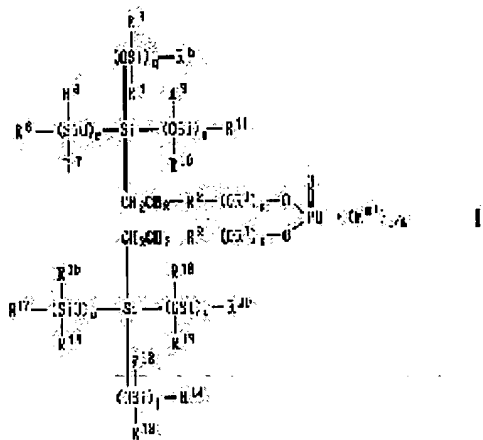
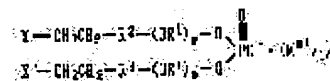
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(54) OILY SOLID COSMETIC

(57)Abstract:

PURPOSE: To obtain a nontacky oily solid cosmetic, good in extensibility and adhesion onto the skin, stable with time without causing liquid sagging and oil floating which have hitherto been problems in blending a silicone oil therein.

CONSTITUTION: This oily solid cosmetic contains (A) 0.1-10wt.% phosphoric diester polyvalent metallic salt-modified organo(poly)siloxane in which two Si atoms in the organo(poly)siloxane or one Si atom each in two molecules of the organo(poly)siloxane is bound to direct bonds X and X' in a compound of formula I [R¹ is a 2-20C alkylene; R² is a 1-20C alkylene which can be substituted with OH; M is an alkaline earth metal or a bi- or a polyvalent transition metal; (n) is the valence of M; (p) is 0-200; X and X' are the direct bonds], (B) 0.1-30wt.% cosmetic oil which is a liquid or a semisolid at ambient temperature, (C) 1-90wt.% silicone oil that is a liquid at ambient temperature and (D) 5-90wt.% water repellent-treated pigment. A compound of formula II [R³ to R²⁰ are each a 1-22C alkyl, a 1-20C alkoxy or phenyl; (q) to (v) are each 0-1000] is preferred as the ingredient (A).



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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the charge of oily solid makeup excellent also in stability, like there is no exudation of an oil while the spread nature and adhesion on the skin are good and excellent in makeup performances, such as makeup **** and covering force, in more detail about the charge of oily solid makeup.

[0002]

[Description of the Prior Art] Generally the charge of oily solid makeup is a charge of makeup manufactured by making into a major component at this what carried out mixed distribution of the fine particles for the charges of makeup using the oily basis which consists of a liquid oil or a semisolid oil, and a solid-state oil, and the oily basis which made the oily gelling agent blend and gel further, and solidifying and casting this. Moreover, the charge of oily solid makeup has mainly been used abundantly from the compatibility over the skin, the water resistance of a makeup film, etc. being excellent at the product for a makeup.

[0003] Generally, the various charge fine particles of makeup and the oily component contained as a major component, and in order to obtain the feeling of use which was excellent as cosmetics, the fine-particles component and the oily component which have various shape properties have been examined by the charge of oily makeup. In order to use spherical fine particles, such as nylon powder, polystyrene, and a polymethylmethacrylate, as a fine-particles component especially in order to improve spread nature, or to raise the adhesion to the skin, tabular fine particles, such as a mica, are used. Furthermore, in recent years, the charge of oily makeup which blended the poly methyl silsesquioxane powder (JP, 1-268615, A) of spherical cellulose powder (JP, 61-100514, A), boron nitride (JP, 63-100508, A), and a true spherical etc. is also indicated. However, generally it is necessary to blend an oily basis 30% of the weight or more, and in order to be the point of usability or a moldability and to hold circulation-proof intensity in this case further, it is considering as the shape of a solid-state, and the oil gel at the above-mentioned charge of oily makeup using a lot of waxes and oil gelling agents. For this reason, while the spread nature on the skin was inferior, there is a feeling of oiliness and it had the fault of being sticky. Then, in order to improve a feeling of oiliness, and stickiness, when the blending ratio of coal of fine particles was made [many], the fault that spread nature got worse further and adhesion and a skin moisturizincy effect also got worse was seen.

[0004] On the other hand, silicon oil, such as dimethylpolysiloxane and annular silicone, does not have a feeling of stickiness, it is positively used in the field of perfumery and cosmetics and cosmetics as an oily high component of safety, and research of technology or use development which harnessed such a property is done briskly.

[0005] However, although silicon oil is desirable as perfumery and cosmetics or an oily medicine for cosmetics, in order to blend with the charge of oily solid makeup, it has the following troubles. That is, compatibility with other oily medicines for cosmetics is bad, it is made to dissolve uniformly, or since the manufacture of a product and stability which used silicon oil as the base are difficult, silicon oil

oozes out easily out of the charge of makeup, and silicon oil has the fault that separation will take place. moreover, the charge of makeup which contains silicon oil so much since ** which still adjusts the viscosity of silicon oil does not exist -- general -- viscosity -- low -- being inferior to stability **** -- the time of use -- liquid -- it had many problems, such as producing whom Furthermore, when silicon oil was used as an oily medicine, while composing the matter with a specific gravity difference, it also had the fault that it was difficult to make it distribute stably with time.

[0006] Then, in order to solve this fault, silicon oil is made to gel, and the attempt used as the perfumery and cosmetics which employed the property efficiently, or a cosmetics basis is made (for example, JP,63-152308,A, JP,1-190757,A, JP,1-201354,A, etc.).

[0007] however, CHIKISOTORO in connection with the usability and the feeling of use which are an important element in perfumery and cosmetics or cosmetics although each of these methods is insoluble to silicon oil as a gelling agent and the stability of gel is good using the organopolysiloxane polymerization object swollen in silicon oil as for these -- it had the fault that a pick rheology property was missing

[0008]

[Problem(s) to be Solved by the Invention] therefore -- without this invention spoils the advantage in the charge of oily solid makeup which blended silicon oil as gel -- CHIKISOTORO -- it aims at offering the charge of oily solid makeup which also served as and offered the pick rheology property

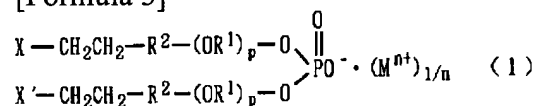
[0009]

[Means for Solving the Problem] In such the present condition, as a result of this invention persons' inquiring wholeheartedly, the specific ORGANO (poly) siloxane which denaturalized by phosphoric-acid diester polyvalent metallic salt harnesses the property of silicon oil. It is the gelling agent which can give a pick rheology property, and the spread nature and adhesion on the skin are good by blending this. and CHIKISOTORO -- It found out that the charge of oily solid makeup which was excellent in the stability which is excellent in makeup performances, such as makeup **** and covering force, and does not have the exudation of an oil was obtained, and this invention was completed.

[0010] namely, this invention -- two silicon atoms in a following component (a), (b), (c), and (d):(a) ORGANO (poly) siloxane -- or the dyad ORGANO (poly) siloxane -- respectively -- one silicon atom -- the following general formula (1)

[0011]

[Formula 3]



[0012]

(M shows alkaline earth metal or the transition metals more than divalent, n shows the valence of M for the alkylene machine of the straight chain of the carbon numbers 1-20 by which, as for R², the hydroxy group may replace the alkylene machine of the straight chain of carbon numbers 2-20, or branched chain among a formula in R¹, or branched chain, p shows the number of 0-200, and X and X' shows a joint hand)

The phosphoric-acid diester polyvalent-metallic-salt denaturation ORGANO (poly) siloxane which comes out, combines with the joint hand X of a phosphoric-acid diester polyvalent-metallic-salt machine and X' which are expressed, and is embellished At the (b) room temperature, 0.1 to 10% of the weight A liquid, or semisolid-like the oily medicine for the charges of makeup At the (c) room temperature, 0.1 to 30% of the weight The silicon oil of a liquid 1 - 90 % of the weight, (d) water-repellent-finish pigment The charge of oily solid makeup characterized by containing 5 - 90 % of the weight is offered.

[0013] In the phosphoric-acid diester polyvalent-metallic-salt machine expressed with the formula (1) in the phosphoric-acid polyvalent-metallic-salt denaturation ORGANO (poly) siloxane of the component (a) in this invention R¹ The straight chain of carbon numbers 2-20 or branched chain preferably as an alkylene machine of a straight chain Ethylene, a propylene, trimethylene, a butylene, pentamethylene, A

hexamethylene, heptamethylene, an octamethylene, nonamethylene, A deca methylene, undecamethylene, a dodeca methylene, a trideca methylene, A tetrapod deca methylene, a PENTA deca methylene, a hexadecamethylene machine, etc. are mentioned. The hexamethylene of carbon numbers 6-12, heptamethylene, an octamethylene, nonamethylene, a deca methylene, undecamethylene, a dodeca methylene group, etc. are mentioned more preferably, and an octamethylene, nonamethylene, and a deca methylene group are desirable also especially in this.

[0014] Moreover, R2 As an alkylene machine of the straight chain of the carbon numbers 1-20 which the hydroxy group may replace, or branched chain A methylene, ethylene, trimethylene, a propylene, 1-methyl propylene, Butylene, pentamethylene, 3-methyl butylene, 1, and 1-dimethyl propylene, A hexamethylene, an octamethylene, nonamethylene, a deca methylene, undecamethylene, 2-hydroxy ethylene, a 2-hydroxy butylene, a 2-hydroxy octamethylene machine, etc. are desirable, and the methylene of carbon numbers 1-10, ethylene, a propylene, a butylene, pentamethylene, a hexamethylene, an octamethylene machine, etc. are mentioned. It is especially R2. p R1 That from which the number of total carbons is set to 3-12 is desirable.

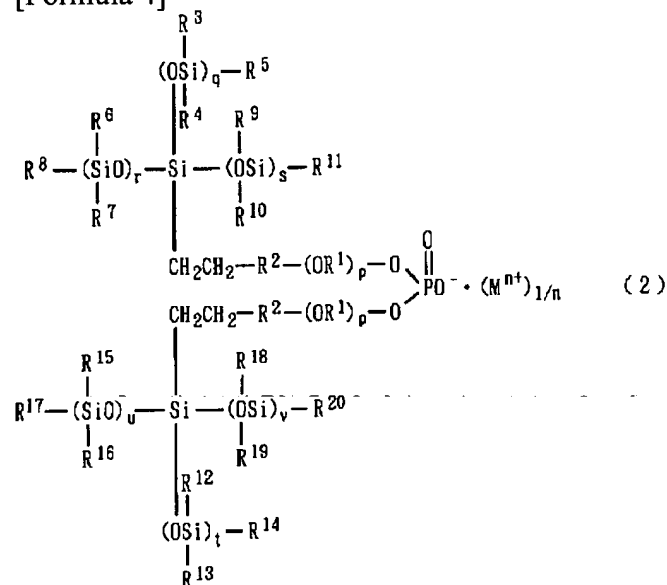
[0015] Moreover, as an alkaline earth metal of M, Mg, calcium, Ba, etc. are mentioned, Mn, Fe, Co, aluminum, nickel, Cu, V, Mo, Nb, etc. are mentioned as transition metals more than divalent, things desirable [among these] are calcium, aluminum, and Fe, and especially its calcium and aluminum are desirable. In addition, n is the valence of M.

[0016] Although p shows the number of 0-200, its number of 0-10 is desirable, and especially its number of 0-5 is desirable.

[0017] In this invention, the phosphoric-acid diester polyvalent-metallic-salt denaturation ORGANO (poly) siloxane expressed with the following formula (2) is mentioned as a desirable thing of the phosphoric-acid diester polyvalent-metallic-salt denaturation ORGANO (poly) siloxane of a component (a).

[0018]

[Formula 4]



[0019] Or it differs. the inside of a formula, q R3, and R4 and R -- 5 or r R6 and R7 and R8, s R9 and R10 and R11, t R12 and R13 and R14, u R15 and R16 and R17, v R18 and R19, and R20 are the same respectively -- The alkyl group, alkoxy group, or phenyl group of the straight chain of carbon numbers 1-22 or branched chain is shown, and q, r, s, t, u, and v show the number of respectively the same or differences 0-1000. However, it is R8 when all of q, r, s, u, and v are set to 0. R17 makes a ring through a divalent oxygen atom. R1, R2, and M, n and p are the same as the above.

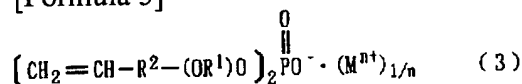
[0020] As an alkyl group of R3-R20 in a formula (2), the alkyl group of carbon numbers 1-22 is

desirable, for example, a methyl, ethyl, a propyl, an isopropyl, butyl, a hexyl, a pentyl, t-butyl, an octyl, tetradecyl, DOKOSHIRU, an octadecyl machine, etc. are mentioned, and especially a methyl group is desirable. As an alkoxy group, the alkoxy group of carbon numbers 1-22 is desirable, for example, methoxy and ethoxy ** propoxy, pentyloxy one, butoxy one, 2-ethyl butoxy, 2-ECHIRUHEKI siloxy machine, etc. are mentioned, and especially a methoxy and an ethoxy basis are desirable.

[0021] The phosphoric-acid diester polyvalent-metallic-salt denaturation ORGANO (poly) siloxane of a component (a) is for example, the following formula (3).

[0022]

[Formula 5]

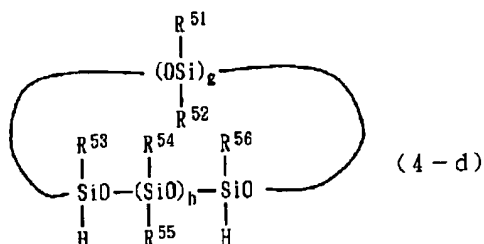
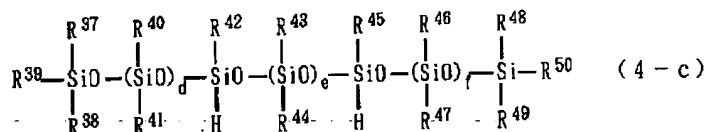
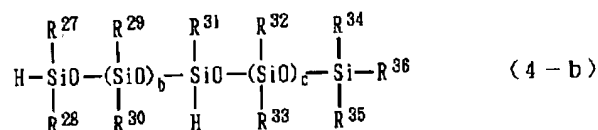
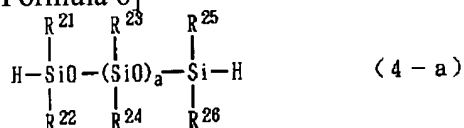


[0023] To the unsaturation machine content phosphoric-acid diester polyvalent metallic salt of one mol expressed with (R1, R2, M, n, and p show the same thing as the above among a formula) The ORGANO hydrogen (poly) siloxane (4) 1 mol which has two silicon atoms combined with a hydrogen atom in the ORGANO (poly) siloxane, Or it can manufacture by making two mols (4') of the ORGANO hydrogen (poly) siloxanes which have one silicon atom combined with a hydrogen atom react.

[0024] As an ORGANO hydrogen (poly) siloxane (4) which has two silicon atoms combined with a hydrogen atom among the raw materials in the above-mentioned method for example, the following formula (4-a) - (4-d) a thing -- moreover, as an ORGANO hydrogen (poly) siloxane (4') which has one silicon atom combined with a hydrogen atom, the following formula (4'-a) - (4'-d) a thing are mentioned, for example

[0025]

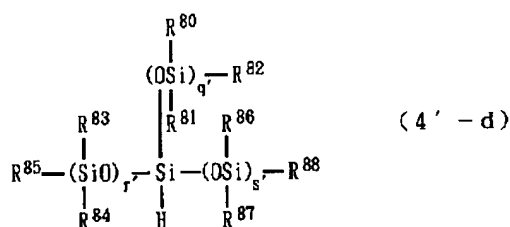
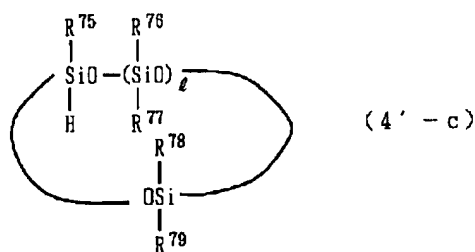
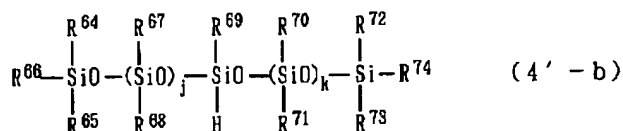
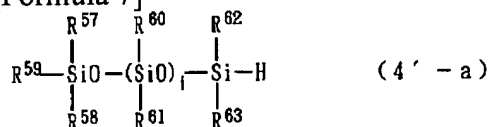
[Formula 6]



[0026] (Among a formula) R21, R22, a R23 And R24, R25, R26, R27, R28, b R29 and R30 and R31, c R32 and R33, R34, R35, R36, R37, R38 and R39, d R40 and R41 and R42, e R43 and R44 and R45, f R46 And R47, R48, R49, R50, g R51 and R52 and R53, h R54 and R55, and that R56 is the same respectively or a difference, The same thing as the above R3-R20 is shown, and a, b, c, d, e, f, g, and h show respectively the same or a difference, and the number of 0-1000.

[0027]

[Formula 7]



[0028] (Among a formula) R57, R58, R59, i R60 And R80 of R70 and R71, R72, R73, R74 and R75, and j R76 and R77, R78 and R79 and q' individuals and R81 and R82, r R83 [R67 and R68 and R69, and] [R61, R62, R63, R64, R65, R66, and] [k] [l] And what has R84, R85, R86 and R87 of s' individual, and R88 same as a difference and the above R3-R20 is shown, and i, j, k, l, q', r', and s' show the number of 0-1000. [respectively same or]

[0029] When a formula (4'-d) is independently used among these raw materials, that whose all of q', r', and s' are not 0 is desirable.

[0030] These ORGANO hydrogen (poly) siloxanes (4) and (4') as an example For example, 1, 3, 5, 7, 9-pen reservoir chill cyclopentasiloxane, 1, 3, 5, 7-tetramethyl cyclotetrasiloxane, 1, 1, 1, 3, 5, 7 and 7, 7-octamethyl tetrapod siloxane, A tris (trimethylsiloxy) silane, 1, 1, 3, 3 and 5, 5-hexa methyl trisiloxane, 1, 1, 1, 3, 5, 5, and 5-heptamethyl trisiloxane, pentamethyldisiloxane, 1, 1, 3, and 3-tetramethyl disiloxane, 1, 3-diphenyl -1, 3-dimethyl disiloxane, 1, 1, 1, 3, 3, 5, and 5-heptamethyl trisiloxane, a methyl tris (dimethyl siloxy) silane, 1, 1, 3, 3, 5, 5 and 7, 7-octamethyl tetrapod siloxane, etc. can be mentioned.

[0031] In this invention, although what was manufactured by the well-known method can be used for this ORGANO hydrogen (poly) siloxane, commercial elegance can also be used as it is and TSF484, TSF483, XF40-A2606, XF40-A1629, XF40-A5149, XF40-A2484 (above, Toshiba Silicone make), etc. are mentioned as the example.

[0032] Moreover, what it could be manufactured by the well-known method, could be manufactured by what process, and was manufactured by the following method can be used for the unsaturation machine content phosphoric-acid diester polyvalent metallic salt expressed with the general formula (3) used for the manufacturing method of a component (a). First Namely, a tetrahydrofuran [alcohol / [CH₂=CH-R₂-(OR₁) p-OH; R₁, R₂, and p show the same meaning as the above] / that has a unsaturated bond at the

end of a mol two to 2.5 times preferably / oxy-halogenation Lynn the two to 4 time mol of this, and], The phosphoric-acid diester which reacts under existence of catalysts, such as a triethylamine, tributylamine, and a pyridine, in solvents, such as diethylether, a methylene chloride, and toluene, is subsequently made to react at 0-10 degrees C by -30--10 degree C for 2 to 12 hours for 2 to 4 hours, and corresponds is obtained. Furthermore, this thing, As opposed to this The 1/-n to 10 time equivalent (n the same meaning as the above) Shown calcium (CH₃COO) The polyvalent metallic salt of 2, CaCl₂, CaBr₂ and CaCO₃, aluminum₂(SO₄)₃, aluminum (CH₃COO)₃, AlCl₃, aluminum (NO₃)₃, BaCO₃, Fe (ClO₄)₃, Fe (NO₃)₃ and FeCl₃, and Mn(CH₃COO)₂ grade If it is made to react at 20-70 degrees C in the mixed solvent of solvents, such as a tetrahydrofuran, ethanol, a methanol, and a butanol, and water and recrystallization, washing, etc. subsequently refine if needed, unsaturation machine content phosphoric-acid diester polyvalent metallic salt (3) will be obtained.

[0033] the ORGANO hydrogen (poly) siloxane (4) described above in order to have enforced the above-mentioned method -- what is necessary is just to make the unsaturation content phosphoric-acid diester polyvalent metallic salt expressed with a general formula (3) react at 20-100 degrees C with or (4') for 1 to 10 hours in the solvent which may dissolve both raw materials, such as toluene of the 1 - 100 time weight of a raw material total amount, a hexane, chloroform, a diisopropyl ether, and a tetrahydrofuran

[0034] In addition, in this reaction, it is the purpose which promotes a reaction and it is desirable that a raw material total amount carries out mol addition of the catalysts, such as a compound of transition metals, such as platinum, a rhodium, nickel, and palladium, and these transition metals and a complex of these transition metals, 10-4 to ten to 6 times.

[0035] After a reaction end, if a solvent is distilled off and acetone extractives are subsequently removed after adding and filtering activated carbon etc., in order to remove a catalyst, the phosphoric-acid diester polyvalent-metallic-salt denaturation ORGANO (poly) siloxane of the component (a) in this invention will be obtained.

[0036] The phosphoric-acid diester polyvalent-metallic-salt denaturation ORGANO (poly) siloxane obtained in this way acts as a gelling agent in this invention. As for especially the loadings of the component (a) to the charge of this invention makeup, it is desirable to blend 0.5 to 8% into the whole quantity 0.1 to 10% of the weight (for it to only be hereafter indicated as "%"). At less than 0.1%, if the charge of oily solid makeup of this invention cannot be stabilized and it exceeds 10%, the viscosity of the charge of makeup will become remarkably high, the mileage at the time of use is bad, and since a feeling of oiliness and a feeling of stickiness are given, it is not desirable.

[0037] As a liquid, or semisolid-like an oily medicine for the charges of makeup, the oily medicine used widely by charges of makeup, such as synthetic ester oil of a multi-wax, a candelilla low, yellow bees wax, a cull navarho, haze wax, a rice bran low, paraffin wax, a liquid paraffin, vaseline, squalane, olive oil, lanolin, higher alcohol, a fatty acid, and a higher alcohol and a fatty acid, for example is mentioned at the room temperature of the component (b) in this invention, and these can also be used combining two or more sorts,

[0038] As for especially the loadings of the component (b) to the charge of this invention makeup, it is desirable to blend 0.5 to 20% 0.1 to 30% into the whole quantity. At less than 0.1%, if the charge of oily solid makeup of this invention cannot be stabilized and it exceeds 30%, the viscosity of the charge of makeup will become remarkably high, the mileage at the time of use is bad, and since a feeling of oiliness and a feeling of stickiness are given, it is not desirable.

[0039] At the room temperature of the component (c) in this invention, as silicon oil of a liquid, dimethylpolysiloxane, a dimethyl cyclo polysiloxane, a methylphenyl polysiloxane, etc. are mentioned, for example, and these can also be used combining two or more sorts, even if independent.

[0040] 5 - 30% of especially the loadings of the component (c) to the charge of this invention makeup are desirable 1 to 90% in the whole quantity. At less than 1%, since a feeling of oiliness will become high if the feel of silicon oil is not discovered and it exceeds 90%, it is not desirable.

[0041] In the water-repellent-finish pigment of the component (d) in this invention As mother fine particles given a water-repellent finish, for example, talc, a mica, a kaolin, a sericite, A muscovite, a synthetic mica, a phlogopite, a red mica, a biotite, a lithia mica, a vermiculite, A magnesium carbonate,

a calcium carbonate, ** SOU soil, a magnesium silicate, A calcium silicate, aluminum silicate, silicic acid barium, silicic acid strontium, A tungstic-acid metal salt, a hydroxyapatite, water silicic acid, a silicic anhydride, A magnesium oxide, a bentonite, a zeolite, ceramics powder, Inorganic fine particles, such as a barium sulfate, an aluminum oxide, an aluminum hydroxide, and boron nitride; Nylon powder, Polyethylene powder, the poly methyl benzoguanamine powder, polymethylmethacrylate powder, Polytetrafluoroethylene powder, JISUCHIREMBENZEN pinhole polymer powder, Organic fine particles, such as microcrystalline cellulose; Titanium oxide, a zinc oxide, a zirconium oxide, An iron oxide (red ocher), titanac-acid iron, an iron hydroxide, ocher, a black iron oxide, carbon black, Mango violet, cobalt violet, a chrome oxide, chromium hydroxide, Inorganic coloring fine particles, such as cobalt titanium, ultramarine blue, and Berlin blue; A titanium oxide coating mica, A titanium oxide coating bismuth oxychloride, a bismuth oxychloride, Pearl pigments, such as titanium oxide coating talc, a scales foil, and a coloring titanium oxide coating mica; Aluminum powder, Metal-powder pigments, such as stainless steel powder and kappa powder; Red No. 3, Red No. 104, red No. 106, red No. 201, red No. 202, red No. 204, Red No. 205, red No. 220, red No. 226, red No. 227, red No. 228, Red No. 230, red No. 401, red No. 505, yellow No. 4, yellow No. 5, Yellow No. 202, yellow No. 203, yellow No. 204, yellow No. 401, blue No. 1, Blue No. 2, blue No. 201, blue No. 404, green No. 3, green No. 201, Green No. 204, green No. 205, orange No. 201, orange No. 203, orange No. 204, Organic pigment fine particles, such as coloring matter; tar coloring matter; carminic acids, such as orange No. 206 and orange No. 207, a laccainic acid, cull SAMIN, brazilin, and a crocin; the insoluble matter is substantially mentioned to water and oils, such as a zirconia, barium, or aluminium lake organic pigment fine particles.

[0042] As the method of a water-repellent finish, for example, fats and oils can be made to be able to stick to a mother fine-particles front face, or the siliconizing method using the fats-and-oils approach which is made to start the esterification and the etherification using functional groups, such as a hydroxyl group, and makes mother fine particles lipophilic property, the metallic-soap approach using zinc salt and magnesium salt of a fatty acid, dimethylpolysiloxane, or the methyl hydrogen polysiloxane, the method of processing with the fluorine compound which has a perfluoroalkyl machine, etc. can be mentioned.

[0043] As for especially the loadings of the component (d) to the charge of this invention makeup, it is desirable to blend 10 to 70% 5 to 90% into the whole quantity. Less than 5% of the covering force is insufficient, and since a feeling of oiliness will be lost if it exceeds 90%, it is not desirable.

[0044] Moreover, it is a book about the arbitrary component further blended into the charge of oily solid makeup of this invention at general charges of makeup, such as a surfactant, a water soluble polymer, antiseptics, a medicine, perfume, a moisturizer, a thickener, and water, if needed.

[0045] As this arbitrary component, specifically Polyoxyethylene alkyl ether, Polyoxyethylene fatty acid ester, polyoxyethylene sorbitan fatty acid ester, A glycerine fatty acid ester, polyglyceryl fatty acid ester, polyoxyethylene glycerine fatty acid ester, Nonionic surface active agents, such as polyoxyethylene hydrogenated castor oil and polyoxyethylene sorbitol fatty acid ester; A sodium stearate, The anionic surface active agent; cationic surface active agent; amphoteric surface active agent represented with fatty-acid soap, such as a palmitic-acid triethanolamine : A carboxymethyl cellulose, A methyl cellulose, a hydroxymethyl cellulose, polyvinyl alcohol, A polyvinyl pyrrolidone, a tragacanth gum, a carrageenan, locust bean rubber, A dextrin, dextrin fatty acid ester, a carboxyvinyl polymer, Water soluble polymers, such as xanthene gum, gelatin, a sodium alginate, and gum arabic : A sorbitol, Xylitol, a glycerol, a maltitol, a propylene glycol, 1, 3-butylene glycol, 1, 4-butylene glycol, pyrrolidone carboxylic-acid sodium, Antiseptics, such as moisturizer:PARAOKISHI benzoic-acid alkyl ester, such as a lactic acid, a sodium lactate, and a polyethylene glycol, a sodium benzoate, and a sorbic acid potassium salt: Medicines, such as vitamins, a vegetable drug, an antiphlogistic, and a germicide, etc. are mentioned.

[0046] Let the charges of oily solid makeup of this invention be the charges of makeup makeup and the charges of basic makeup, such as foundation, a lip stick, and rouge, by the conventional method.

[0047]

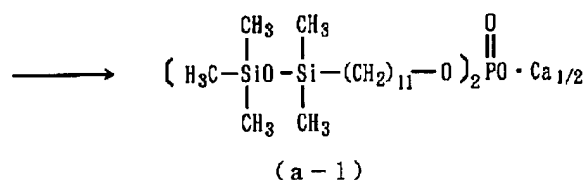
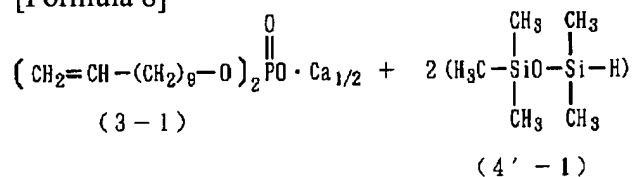
[Effect of the Invention] the silicon oil of a feeling of use, safety, and transparency with the high charge of oily solid makeup of this invention -- CHIKISOTORO -- the liquid which had become a problem when silicon oil was blended, while the spread nature and adhesion on the skin were good and were non-adhesiveness, since the gel which gives a pick rheology property and moreover does not have stringiness was built -- who and an oil float are not raised and it has with time the feature of being stable

[0048]

[Example] Hereafter, although an example, a synthetic example, and the example of an examination explain this invention concretely, this invention is not limited to these.

[0049] The synthetic example 1. [0050]

[Formula 8]



[0051] 15g (3-1) of compounds was dissolved in toluene 500g, and 11g (4'-1) (Shin-Etsu Chemical Co., Ltd. make) of compounds and the toluene 30g solution were added in this solution. The chloroplatinic acid (isopropyl alcohol solution) was added to this solution, and it was made to ripe at 70 degrees C for 8 hours. After [a reaction end] methanol 100g and 1g of activated carbon were added, and it stirred at 30 degrees C for 1 hour. After filtering activated carbon, reduced pressure distilling off of the solvent was carried out. After adding chloroform 100g to the residue and dissolving a product, the solid-state which added acetone 500g and was produced was filtered, and 23g (a-1) of compounds was obtained. IR(KBr): Si-O-Si, 1070cm-11 H-NMR(CDC13):-0.1 ppm Si-CH3, 0.4 ppm Si-CH2, 1.2 ppm -CH2-, 1.6ppm-(CH2)-CH2 O-P, 3.8ppm-CH2-CH2 O-P. [0052]

[Formula 9]

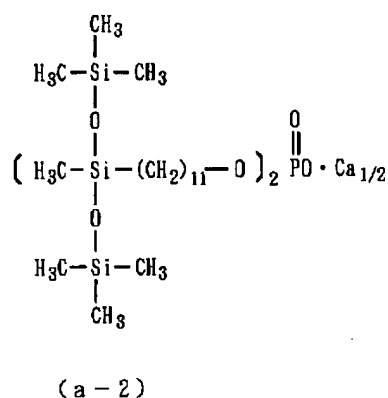
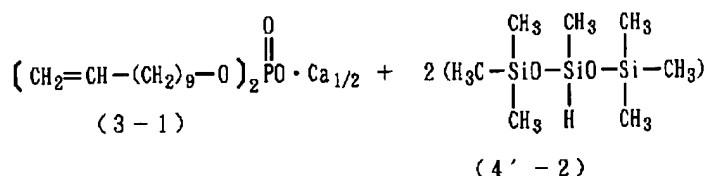
^{31}P -NMR(CDC ℓ_3) :

-1.72ppm



[0053] The synthetic example 2. [0054]

[Formula 10]

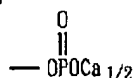


[0055] 10g (3-1) of compounds was dissolved in toluene 400g, and 11g (4'-2) (Shin-Etsu Chemical Co., Ltd. make) of compounds and the toluene 30g solution were added in this solution. The chloroplatinic acid (isopropyl alcohol solution) was added to this solution, and it was made to ripe at 70 degrees C for 8 hours. After [a reaction end] methanol 100g and 1g of activated carbon were added, and it stirred at 30 degrees C for 1 hour. After filtering activated carbon, reduced pressure distilling off of the solvent was carried out. After adding chloroform 100g to the residue and dissolving a product, the solid-state which added acetone 450g and was produced was filtered, and 20g (a-2) of compounds was obtained. IR(KBr): Si-O-Si, 1060cm-11 H-NMR(CDC13):-0.1, -0.2 ppm Si-CH3, 0.4 ppm Si-CH2, 1.2ppm-CH2-, 1.5ppm-(CH2)-CH2 O-P, 3.8ppm-CH2-CH2 O-P. [0056]

[Formula 11]

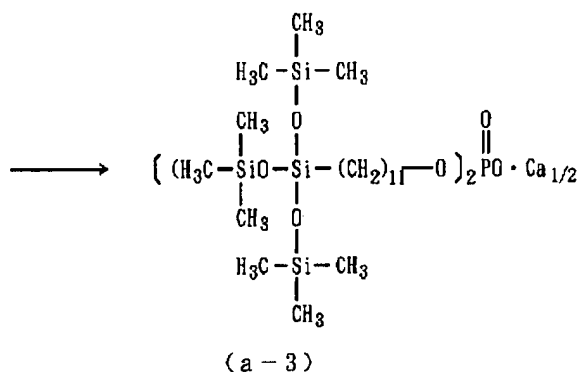
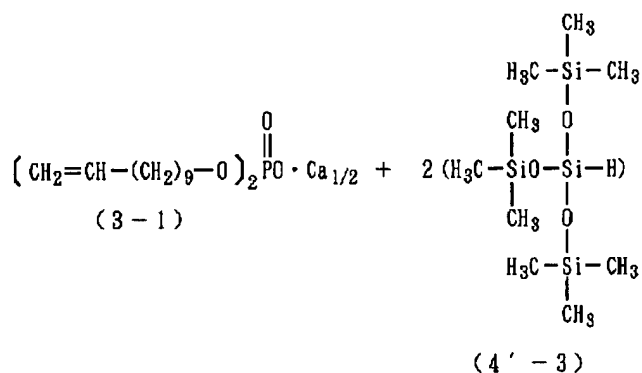
^{31}P -NMR(CDC ℓ_3) :

-1.74ppm



[0057] The synthetic example 3. [0058]

[Formula 12]

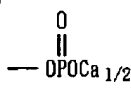


[0059] 10g (3-1) of compounds was dissolved in toluene 400g, and 15g (4'-3) (Shin-Etsu Chemical Co., Ltd. make) of compounds and the toluene 40g solution were added in this solution. The chloroplatinic acid (isopropyl alcohol solution) was added to this solution, and it was made to ripe at 70 degrees C for 8 hours. After [a reaction end] methanol 100g and 1g of activated carbon were added, and it stirred at 30 degrees C for 1 hour. After filtering activated carbon, reduced pressure distilling off of the solvent was carried out. After adding chloroform 100g to the residue and dissolving a product, the solid-state which added acetone 550g and was produced was filtered, and 23g (a-3) of compounds was obtained. IR(KBr): Si-O-Si, 1070cm-1 1 H-NMR(CDC13):-0.1 ppm Si-CH3, 0.4 ppm Si-CH2, 1.2ppm-CH2-, 1.5ppm-(CH2)-CH2 O-P, 3.8ppm-CH2-CH2 O-P. [0060]

[Formula 13]

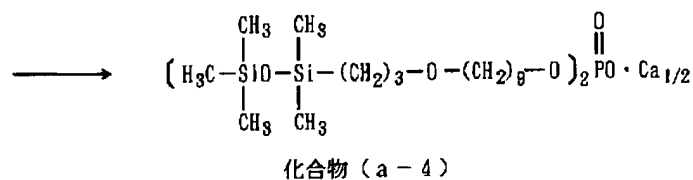
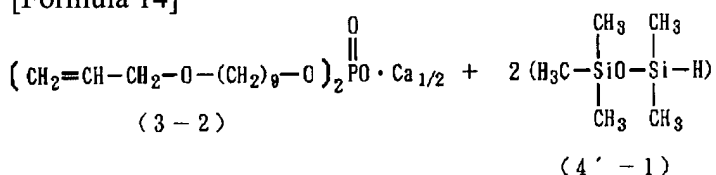
^{31}P -NMR(CDC ℓ_3) :

-1.84ppm



[0061] The synthetic example 4. [0062]

[Formula 14]

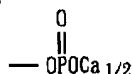


[0063] 10g (3-2) of compounds was dissolved in toluene 300g, and 7g (4'-1) (Shin-Etsu Chemical Co., Ltd. make) of compounds and the toluene 40g solution were added in this solution. The chloroplatinic acid (isopropyl alcohol solution) was added to this solution, and it was made to ripe at 70 degrees C for 8 hours. After [a reaction end] methanol 100g and 1g of activated carbon were added, and it stirred at 30 degrees C for 1 hour. After filtering activated carbon, reduced pressure distilling off of the solvent was carried out. After adding chloroform 100g to the residue and dissolving a product, the solid-state which added acetone 550g and was produced was filtered, and 15g (a-4) of compounds was obtained. IR(KBr): Si-O-Si, 1057cm-1 H-NMR(CDC13):-0.1 ppm Si-CH3, 0.4 ppm Si-CH2, 1.2ppm-CH2-, 1.5ppm-(CH2)-CH2 O-P, -(CH2)-CH2-O, 3.3ppm-CH2-O-CH2-, 3.8ppm-CH2-CH2 O-P. [0064]

[Formula 15]

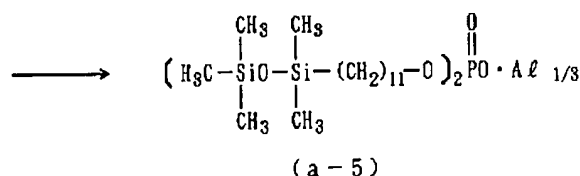
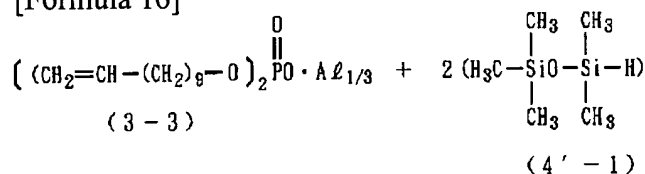
^{31}P -NMR(CDC ℓ_3) :

-1.74ppm



[0065] The synthetic example 5. [0066]

[Formula 16]



[0067] 8.0g (3-3) of compounds was dissolved in toluene 250g, and 6.5g (4'-1) (Shin-Etsu Chemical Co., Ltd. make) of compounds and the toluene 20g solution were added in this solution. The chloroplatinic acid (isopropyl alcohol solution) was added to this solution, and it was made to ripe at 70 degrees C for 7 hours. After [a reaction end] methanol 100g and 0.5g of activated carbon were added, and it stirred at 30 degrees C for 1 hour. After filtering activated carbon, reduced pressure distilling off of the solvent was carried out. After adding chloroform 100g to the residue and dissolving a product, the solid-state which added acetone 500g and was produced was filtered, and 13g (a-5) of compounds was obtained.

IR(KBr): Si-O-Si, 1047cm-1 H-NMR(CDC13+CD3OD):-0.1 ppm Si-CH3, 0.45 ppm Si-CH2, 1.23ppm-CH2-, 1.5 ppm P-O-CH2-CH2, 3.9 ppm P-O-CH2. [0068]

[Formula 17]

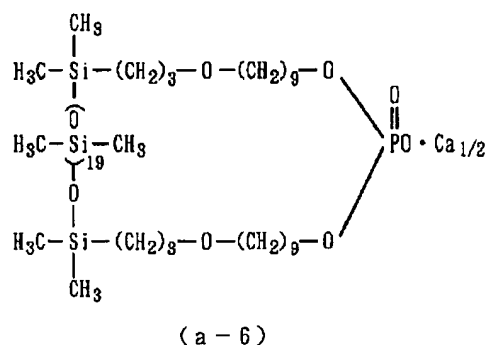
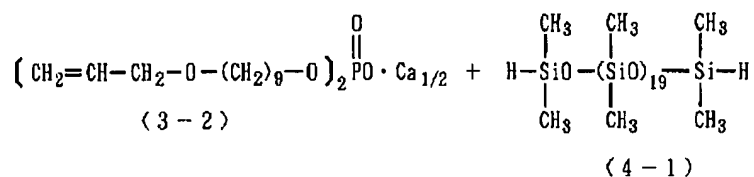
^{31}P -NMR(CDC ℓ_3 +CD $_3$ OD) :

-14.8ppm



[0069] The synthetic example 6. [0070]

[Formula 18]



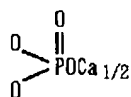
[0071] 5g (3-2) of compounds was dissolved in toluene 100g, and 16g (4-1) (Toshiba Silicone make) of compounds and the toluene 100g solution were added in this solution. The chloroplatinic acid (isopropyl alcohol solution) was added to this solution, and it was made to ripe at 70 degrees C for 8 hours. After [a reaction end] methanol 100g and 1g of activated carbon were added, and it stirred at 30 degrees C for 1 hour. After filtering activated carbon, reduced pressure distilling off of the solvent was carried out. After adding chloroform 150g to the residue and dissolving a product, the solid-state which added acetone 600g and was produced was filtered, and 20g (a-6) of compounds was obtained.

IR(KBr):Si-O-Si, 1090cm⁻¹ H-NMR(CDCl₃+CD₃OD):0 ppm Si-CH₃, 0.46 ppm Si-CH₂, 1.2ppm-CH₂-, and 1.5ppm-CH₂-CH₂-O-P, - CH₂-CH₂-O-CH₂-CH₂-, 3.3ppm-CH₂-O-CH₂-, 3.8ppm-CH₂-CH₂-O-P. [0072]

[Formula 19]

³¹P-NMR(CDCl₃+CD₃OD) :

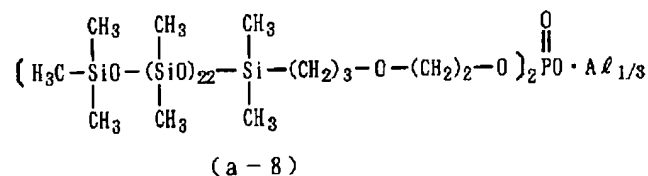
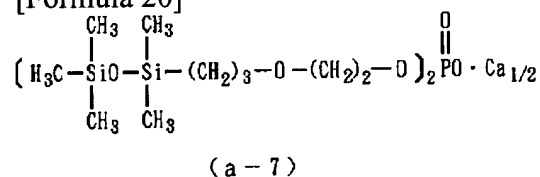
-1.42ppm



[0073] According to the same method as the synthetic example 7 and the examples 1-6 of 8 composition, the compound (a-7) and compound (a-8) which are expressed with the following formula were compounded.

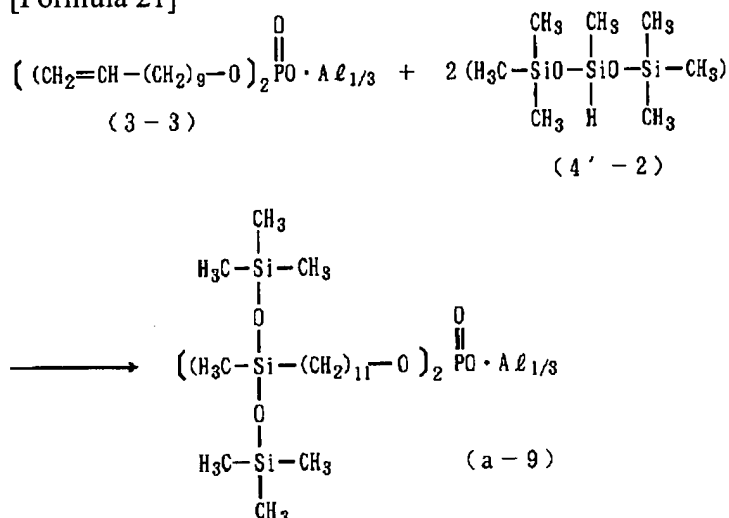
[0074]

[Formula 20]



[0075] The synthetic example 9. [0076]

[Formula 21]

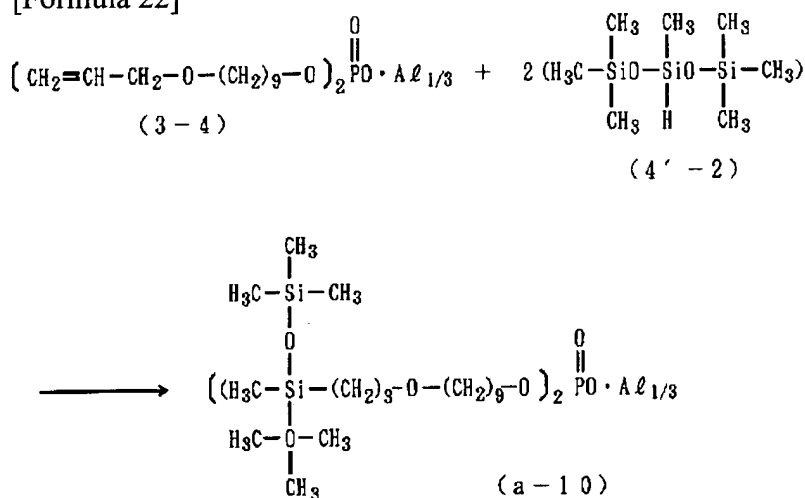


[0077] 7g (3-3) of compounds was dissolved in toluene 150g, and 7.8g (4'-2) (Shin-Etsu Chemical Co., Ltd. make) of compounds and the toluene 10g solution were added in this solution. The chloroplatinic acid (isopropyl alcohol solution) was added to this solution, and it was made to ripe at 70 degrees C for 7 hours. After [a reaction end] ethanol 100g and 0.5g of activated carbon were added, and it stirred at 30 degrees C for 1 hour. After filtering activated carbon, reduced pressure distilling off of the solvent was carried out. The solid-state which added back acetone 400g in which chloroform 50g was added to the residue and the product was dissolved, and was produced was filtered, and 13.8g (a-9) of compounds was obtained.

IR(KBr): Si-O-Si, 1020cm-11 H-NMR(CDC13):-0.1ppm-CH2-Si-CH3, 0ppm-O-Si-CH3, 0.40ppm-CH2-Si, 1.21ppm-CH2-, 1.49ppm-(CH2)-CH2-O-P, 3.90ppm-CH2-O-P31 P-NMR(CDC13):-14.74ppm.

[0078] The synthetic example 10. [0079]

[Formula 22]



[0080] 10g (3-4) of compounds was dissolved in toluene 150g, and 10g (4'-2) (Shin-Etsu Chemical Co., Ltd. make) of compounds and the toluene 10g solution were added in this solution. The chloroplatinic acid (isopropyl alcohol solution) was added to this solution, and it was made to ripe at 70 degrees C for 7 hours. After [a reaction end] ethanol 100g and 0.5g of activated carbon were added, and it stirred at 30 degrees C for 1 hour. After filtering activated carbon, reduced pressure distilling off of the solvent was carried out. The solid-state which added back acetone 400g in which chloroform 50g was added to the residue and the product was dissolved, and was produced was filtered, and 18.8g (a-10) of compounds was obtained.

IR(KBr):Si-O-Si, 1025cm⁻¹ H-NMR(CDCl₃):-0.15ppm-CH₂-Si-CH₃, 0.01ppm-O-Si-CH₃, 0.36ppm-CH₂-Si, and 1.21 ppm - CH₂-, 1.47ppm-(CH₂)-CH₂-O-P, -O-CH₂-CH₂-, 3.29ppm-CH₂-O-CH₂-, 3.83ppm-CH₂-O-P³¹P-NMR(CDCl₃):-13.95ppm. [0081] Example 1 and oily solid foundation composition . % 1. dimethylpolysiloxane A residue 2. compound (a-1) 0.53. Candelilla wax 3.04. Antiseptics Optimum dose 5. titanium oxide (water-repellent finish) 15.06. red ocher (water-repellent finish) 0.87. Yellow iron oxide (water-repellent finish) 2.58. black iron oxide (water-repellent finish) 0.29. mica (water-repellent finish) 31.510. perfume Minute amount. [0082] The process components 1-4 are heated at 90 degrees C, the mixed dissolution was carried out, components 5-9 were added further, and stirring mixture was fully carried out until it became homogeneity, maintaining at 90 degrees C. After adding the component 10 to this mixture and mixing, oily solid foundation was prepared by filling up a metal dish and cooling. In addition, in the water-repellent finish of a water-repellent-finish pigment, commercial methyl hydrogen polysiloxane (KF-99, Shin-etsu silicone company make) was used as a water-repellent chemical.

[0083] Oily solid foundation was obtained like the example 1 except having made the loadings of example 2 and an oily solid foundation compound (a-1) increase to 8%.

[0084] Example of comparison 1, and oily solid foundation composition . % 1. flow isoparaffin Residue 2. JISECHIRURIN acid aluminum 2.03. Candelilla wax 3.04. Titanium oxide (water-repellent finish) 15.05. Red ocher (water-repellent finish) 0.86. Yellow iron oxide (water-repellent finish) 2.57. black iron oxide (water-repellent finish) 0.28. mica (water-repellent finish) 31.59. antiseptics Proper quantity 10. Perfume Minute amount. [0085] The process components 1-4 are heated at 90 degrees C, the mixed dissolution was carried out, components 5-9 were added further, and stirring mixture was fully carried out until it became homogeneity, maintaining at 90 degrees C. After adding the component 10 to this mixture and mixing, oily solid foundation was prepared by filling up a metal dish and cooling. In addition, a water-repellent finish of a water-repellent-finish pigment was performed by the same method as an example 1.

[0086] Example of comparison 2, and oily solid foundation composition . % 1. dimethylpolysiloxane Residue 2. JISECHIRURIN acid aluminum 2.03. Candelilla wax 3.04. Titanium oxide (water-repellent finish) 15.05. red ocher (water-repellent finish) 0.86. yellow iron oxide (water-repellent finish) 2.57. Black iron oxide (water-repellent finish) 0.28. mica (water-repellent finish) 31.59. antiseptics Proper quantity 10. Perfume Minute amount. [0087] The process components 1-4 were heated at 90 degrees C, and the mixed dissolution was carried out. Furthermore, components 5-9 were added, and stirring mixture was fully carried out until it became homogeneity, maintaining at 90 degrees C. After adding the component 10 to this mixture and mixing, oily solid foundation was prepared by filling up a metal dish and cooling. In addition, a water-repellent finish of a water-repellent-finish pigment was performed by the same method as an example 1.

[0088] "The mileage of makeup", "makeup *****", the "covering force", "the exudation of an oil", and the evaluation examination of "firmness" were performed in accordance with the following error criterion about the oily solid foundation of example of examination 1 examples 1 and 2, and the examples 1 and 2 of comparison. The result is shown in Table 1.

[0089] The subjects of the 20 women of 20-his 50's evaluation of "the mileage of makeup", "makeup *****", and the "covering force" are five stages (it excels very much.). It excels. It can be called neither. It is inferior. It is considerably inferior. Organic-functions evaluation was performed, it reached, and although excelled, the case where they were ** and less than 40% about the case where they are O and less than 60% 40% or more about the case which are 60% or more of all subjects where it excels very much was evaluated as x.

[0090] The oily solid foundation of "the exudation of an oil", the evaluation examples 1 and 2 of "firmness", and the examples 1 and 2 of comparison was saved for one month at 40 degrees C and the air conditioned room of 70% of humidity, what the exudation of an oil is not accepted in about the exudation of an oil was made into O, what is accepted was made into x, and O and the inferior thing were evaluated for what is excellent with visual observation as x about firmness.

[0091]

[Table 1]

評価項目	実施例 1	実施例 2	比較例 1	比較例 2
化粧ののび	△	○	×	×
化粧持ち	△	○	×	×
カバー力	○	○	△	×
油のしみ出し	○	○	○	×
保形性	○	○	○	×

[0092] The oily solid foundation obtained in the examples 1 and 2 was extended compared with the oily solid foundation of the examples 1 and 2 of comparison, and makeup **** and the covering force showed the result very good also about the exudation and firmness of an oil good so that more clearly than a result table 1.

[0093] Example 3 and lip-stick composition . % 1. dimethylpolysiloxane A residue 2. compound (a-4) 8.03. Paraffin wax 20.04. Ceresin 7.05. Antiseptics Proper quantity 6. titanium oxide (water-repellent finish) 15.07. pearl pigment (water-repellent finish) 0.88. red No. 202 2.59. perfume Minute amount.

[0094] The process components 1-4 are heated at 90 degrees C, the mixed dissolution was carried out, and stirring mixture was fully carried out until it became homogeneity, adding components 5-8 further and maintaining at 90 degrees C. After adding the component 8 to this mixture and mixing, degassing was carried out and the lip stick was prepared by slushing into a split mold and cooling. In addition, a water-repellent finish of a water-repellent-finish pigment was performed by the same method as an example 1.

[0095] Example 4 and rouge composition . % 1. dimethylpolysiloxane A residue 2. compound (a-9) 8.03. Paraffin wax 20.04. Antiseptics Proper quantity 5. sericite 30.06. titanium oxide (water-repellent finish) 15.07. red ocher (water-repellent finish) 2.08. yellow iron oxide (water-repellent finish) 1.09. perfume Minute amount. [0096] After heating the process components 1-4 at 90 degrees C, having carried out the mixed dissolution, adding components 5-8 further and mixing for about 5 minutes, it finished further, it ground, the metal dish hay press was carried out with the press machine, and rouge was prepared. In addition, a water-repellent finish of a water-repellent-finish pigment was performed by the same method as an example 1.

[0097] The lip stick and rouge which were obtained in the aforementioned examples 3 and 4 were extended [each / conventionally / elegance], and makeup **** and the covering force showed the result very good also about the exudation and firmness of an oil good.

[Translation done.]